

三井・デュボンフロロケミカル株式会社殿

報 告 書 英 訳

(試験番号：A080559)

英訳対象報告書

FRD903の分解度試験

(試験番号：A080558)

2009年 6月17日

三菱化学メディエンス株式会社

FINAL REPORT

M.C.M. REPORT No. A080558

Ready Biodegradability Test of FRD903

Submitted to :

DU PONT-MITSUI FLUOROCHEMICALS COMPANY, LTD.

Prepared by :

Mitsubishi Chemical Medience Corporation

May 25, 2009

**Ready Biodegradability Test of FRD903
(English version)**

Sponsor : DU PONT-MITSUI FLUOROCHEMICALS COMPANY, LTD.
Study Title : Ready Biodegradability Test of FRD903
Study No. : A080558

This report is the English version of the original, which was written in Japanese. I, the undersigned, hereby declare that this version faithfully reflects the original report to the best of my knowledge.

Translated and Approved by


_____ Date : June 17, 2009

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COMPLIANCE WITH THE GLP STANDARDS

Yokohama Laboratory, Toxicological Science Division,
Medi-Chem Business Segment,
Mitsubishi Chemical Medience Corporation

Sponsor : DU PONT-MITSUI FLUOROCHEMICALS COMPANY, LTD.
Study Title : Ready Biodegradability Test of FRD903
Study No. : A080558

I hereby certify that this study was carried out in accordance with its protocol and with our standard operating procedures, and that the reported results reflect the raw data of the study accurately.

The study described in this report was conducted in compliance with the Good Laboratory Practice(GLP) standards concerning the test facility in which the study of new chemical substance is performed (Yakushokuhatsu No.1121003, Heisei 15.11.17 Seikyoku No.3, Kanpokiatsu No.031121004, November 21, 2003;the latest revision, July 4, 2008).

Sumiko Kawashima, B. Sc.
Study Director

Sealed date: May 25, 2009

QUALITY ASSURANCE STATEMENT

Yokohama Laboratory, Toxicological Science Division,
Medi-Chem Business Segment,
Mitsubishi Chemical Medience Corporation

Sponsor : DU PONT-MITSUI FLUOROCHEMICALS COMPANY, LTD.
Study Title : Ready Biodegradability Test of FRD903
Study No. : A080558

I verify that this study was carried out in accordance with the following Good Laboratory Practice (GLP) and the reported results reflect the raw data of the study.

The Good Laboratory Practice (GLP) standards concerning the test facility in which the study of new chemical substance is performed (Yakushokuhatsu No.1121003, Heisei 15.11.17 Seikyoku No.3, Kanpokiatsu No.031121004, November 21, 2003; the latest revision, July 4, 2008).

The types of inspections and their dates, and the dates the results were reported to management and to the Study Director are listed below.

	Date of Inspection	Date of Reporting to Test Facility Management and Study Director
Protocol :		
Draft protocol	March 13, 2009	March 13, 2009
Protocol	March 19, 2009	March 19, 2009
Amendment 01	March 25, 2009	March 25, 2009
Amendment 02	April 1, 2009	April 1, 2009
Amendment 03	May 13, 2009	May 13, 2009
In-progress study :		
Start of BOD measurement	March 25, 2009	March 25, 2009
Measurement of residual test substance	April 22, 2009	April 22, 2009
Final report :		
Draft report	May 18, 2009	May 19, 2009
Final report	May 25, 2009	May 25, 2009

Quality Assurance Personnel

Yumiko Kashiwagi, B. Sc.

Sealed date : May 25, 2009

INTRODUCTION

1 STUDY TITLE

Ready Biodegradability Test of FRD903 (Study No. A080558)

2 PURPOSE

This study was conducted to evaluate the ready biodegradability of the test substance for notification under the Chemical Substances Control Law of Japan.

3 TEST GUIDELINE

The Test Method Relating to New Chemical Substances <Biodegradability Test of Chemical Substances by Microorganisms> (Yakushokuhatsu No. 1121002, Heisei 15.11.13 Seikyoku No. 2, Kanpokiatsu No. 031121002, November 21, 2003; the latest revision, November 20, 2006).

4 GLP COMPLIANCE

The Good Laboratory Practice (GLP) standards concerning the test facility in which the study of new chemical substance is performed (Yakushokuhatsu No. 1121003, Heisei 15.11.17 Seikyoku No. 3, Kanpokiatsu No. 031121004, November 21, 2003; the latest revision, July 4, 2008).

5 SPONSOR

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9 EXPERIMENTAL SCIENTISTS

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Hirokazu Kobayashi, M. Sc.

Yuri Goto, M. Sc.

10 STUDY PERIOD

Study initiation: March 19, 2009

BOD measurement starting: March 25, 2009

BOD measurement completion: April 22, 2009

Study completion: May 25, 2009

11 STORAGE AND RETENTION OF THE TEST SUBSTANCE AND RECORDS

The followings will be stored in the archives of our laboratory.

- 1) Protocol
- 2) Final report
- 3) Raw data
- 4) Test substance
- 5) Other requirements

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SUMMARY

STUDY TITLE

Ready Biodegradability Test of FRD903 (Study No. A080558)

METHOD

The study was conducted in accordance with the Test Method Relating to New Chemical Substances <Biodegradability Test of Chemical Substances by Microorganisms> (Yakushokuhatsu No. 1121002, Heisei 15.11.13 Seikyoku No. 2, Kanpokiatsu No. 031121002, November 21, 2003 ; the latest revision, November 20, 2006).

Study period : March 19, 2009 to May 25, 2009

Test bottles : Bottle 1 (activity control) : aniline + sludge + basal medium
 Bottle 2 (inoculum blank) : sludge + basal medium
 Bottles 3,4,5 (test suspensions) : test substance + sludge + basal medium
 Bottle 6 (abiotic control) : test substance + water
 (test substance : 100 mg/L ; sludge : 30 mg/L)

Measurements : Biochemical oxygen demand (BOD) (for 28 days)
 Dissolved organic carbon (DOC) (at day 28)
 Residual test substance amount (at day 28)

[measured with high performance liquid chromatograph-mass spectrometer (LC/MS/MS)]

RESULTS

Measured values (day 28)

	Bottle No.				Theoretical
	3	4	5	6	value
BOD ^{*1} , mg	1.1	-0.3	1.6	0.0	27.6
DOC ^{*1} , mg	6.3	6.2	6.6	6.4	6.5
Test substance, mg	29.3	30.9	29.8	30.7	30.0

*1 Values of bottles 3, 4 and 5 are corrected with BOD or DOC value of bottle 2.

Degradabilities (%)

	Bottle No.			average
	3	4	5	
BOD	4	0(-1) ^{*2}	6	3
DOC	2	3	0(-3) ^{*2}	2
Test substance	5	0(-1) ^{*2}	3	3

*2 Where % degradability is calculated to be negative, this value is shown in parentheses.

DISCUSSION

From the degradability results based on the BOD (av. 3%), the DOC (av. 2%) and the residual test substance amount (av. 3%), it is concluded that the test substance is not readily degradable and was not transformed structurally under the conditions of this test.

1 MATERIAL

1.1 Test Substance

1.1.1 Name, structural formula and physico-chemical properties

Name of the new chemical substance (by IUPAC nomenclature)	2,3,3,3-tetrafluoro-2-(heptafluoropropoxy) propanoic acid		
Alternate name	FRD903		
CAS RN	13252-13-6		
Structural formula or rational formula (if both of them are unknown, outline of preparation or manufacturing)	$\text{CF}_3\text{CF}_2\text{CF}_2\text{O}\underset{\text{CF}_3}{\underset{ }{\text{CF}}}\text{COOH}$		
Molecular weight	330.05		
Purity(%) of the new chemical substance subjected to the study	99.6		
Lot number of the new chemical substance subjected to the study	0711FRD036-2		
Name and content of impurities	Unknown: about 0.4%		
Vapor pressure	About 1 mmHg (25°C)		
Solubility in water	Above 20 wt%		
1-octanol/water partition coefficient	-		
Melting point	-		
Boiling point	About 85°C /38 mmHg		
Appearance at room temperature	Colorless and clear liquid		
Stability	-		
Solubility in solvents and the like	Solvent	Solubility	Stability in solvent
	Acetone	Above 25 wt%	-

Above information is provided by the sponsor.

1.1.2 Sample

1)Supplier of sample : DU PONT-MITSUI FLUOROCHEMICALS COMPANY, LTD.

2)Elemental composition :C 21.8%, H 0.3%, O 14.5%, F 63.3%

[Elemental composition was calculated based on the chemical structure of the test substance.]

1.1.3 Stability under the storage conditions

The test substance had been sealed and stored in a desiccator at room temperature in the dark for the duration of the study.

After the exposure period, infrared absorption spectrum of the test substance was measured. The spectrum was consistent with that measured before the exposure period, indicating that the test substance was stable under the storage conditions.

[Figure 1]

1)Apparatus : Infrared spectrophotometer Nicolet iS10, Thermo Fisher Scientific Inc.

1.2 Activated sludge

1)Mixed liquor suspended solid (MLSS) : 2350 mg/L

2)Source : Chemicals Evaluation and Research Institute, Japan

3)Date of receipt : January 15, 2009

2 METHODS

2.1 Test methods

The study was conducted in accordance with the Test Method Relating to New Chemical Substances <Biodegradability Test of Chemical Substances by Microorganisms> (Yakushokuhatsu No. 1121002, Heisei 15.11.13 Seikyoku No. 2, Kanpokiatsu No. 031121002, November 21, 2003; the latest revision, November 20, 2006).

The test substance was exposed to activated sludge in a closed-system oxygen consumption measuring apparatus. The biochemical oxygen demand (BOD) was measured over a 28-day period. After this period, amounts of the dissolved organic carbon (DOC) and residual test substance were measured. The biodegradability of the test substance was evaluated from these results.

2.1.1 Degradability study conditions

Conditions

- | | |
|--------------------|---|
| 1) Temperature | : 25 ± 1°C |
| 2) Exposure period | : 28 days |
| 3) Stirring | : Continuous stirring with magnetic stirrer |
| 4) Test volume | : 300 mL |
| 5) Concentration | : test substance (bottles 3-6) : 100 mg/L |
| | aniline (bottle 1) : 100 mg/L |
| | activated sludge (bottles 1-5) : 30 mg/L |

Test bottle contents :

- Bottle 1 : Activity control (aniline + activated sludge + basal medium)
29.5 µL (30.0 mg) of aniline^{*1} was added to the basal medium^{*2}, then activated sludge was added.
- Bottle 2 : Inoculum blank (activated sludge + basal medium)
Activated sludge was added to the basal medium^{*2}.
- Bottles 3,4,5 : Test suspensions (test substance + activated sludge + basal medium)
30.0 mg of the test substance was added to the basal medium^{*2}, and adjusted the pH to 7.0 with 1N sodium hydroxide aqueous solution, then activated sludge was added.
- Bottle 6 : Abiotic control (test substance + purified water)
30.0 mg of the test substance was added to 300 mL of purified water^{*3}.

^{*1} Reference substance : Guaranteed reagent, Lot No.007X1955

Kanto Chemical Co., Inc.

^{*2} The total volume of the basal medium and the sludge was held fixed at 300 mL.

^{*3} Grade A4, Japanese Industrial Standards (JIS) K0557

2.1.2 BOD measurement

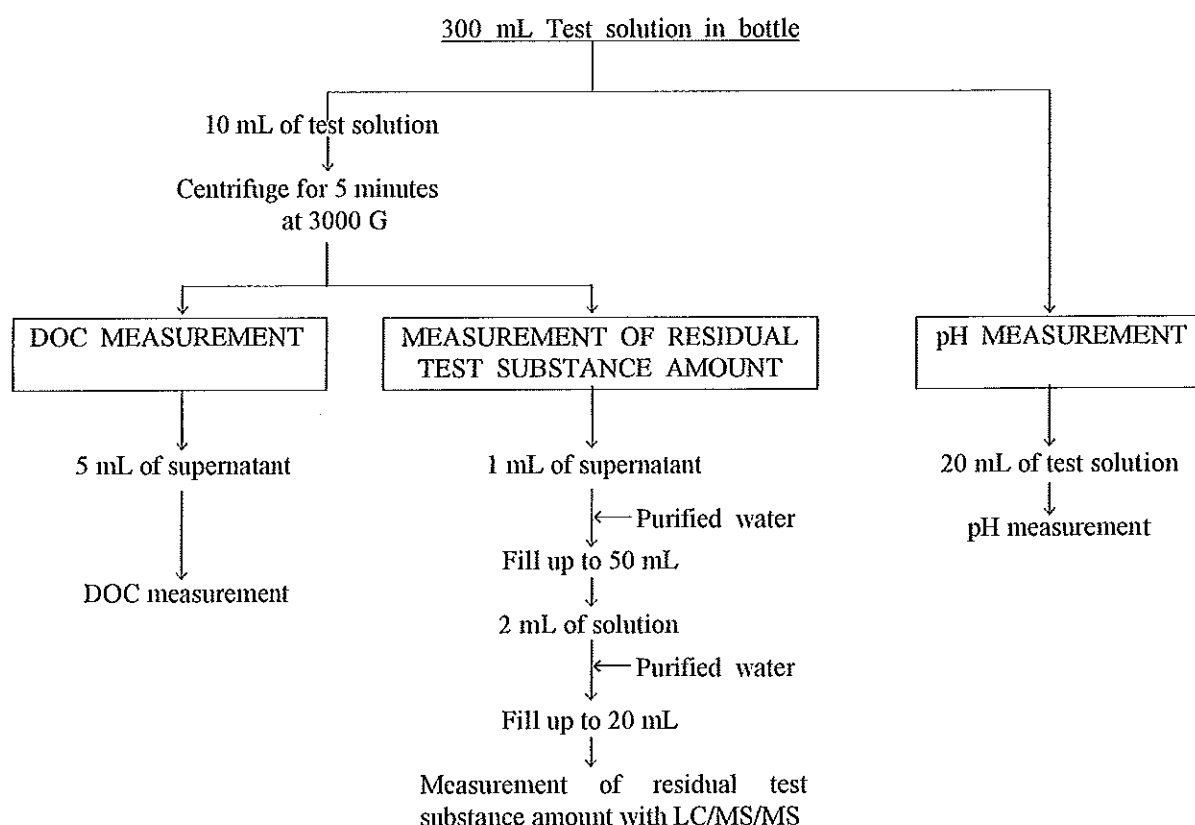
The BOD was measured for 28 days. The test solutions were observed and proper operation of the apparatus was confirmed once a day except holidays throughout the exposure period. After the measurement, the contents in test bottles were observed.

1) Apparatus: Closed system oxygen consumption measuring apparatus

OM-3100A, Ohkura Electric Co. (M.S.I. ID:M)

2.1.3 Pre-treatment of the test solutions after BOD measurement

Pre-treatment of the test solution was performed according to the following flow.



2.1.4 pH measurement

At the start and the end of the exposure period, pH of the test solution was measured.

1) Apparatus : pH meter F-52, HORIBA, Ltd.

2.1.5 DOC measurement

The concentration of the DOC was measured as follows.

1) Apparatus : TOC analyzer TOC-5000A, Shimadzu Co.

2) Conditions : Furnace temperature : 680°C (TC)
 Air flow rate : 150 mL/min
 Sensitivity : $\times 5$
 Injection volume : 30 μ L
 Repeated number : n=3(adopt mean value)

3) Calibration curve

The following standard solutions were injected into the TOC analyzer. The calibration curve was prepared by the data processor of the analyzer.

standard solutions

TC(total carbon): 40 and 80 mg C/L aqueous solutions of potassium hydrogen phthalate.

IC(inorganic carbon): 0 mg C/L purified water.
10 mg C/L aqueous solution of sodium hydrogen carbonate and sodium carbonate.

2.1.6 Measurement of the residual test substance amount

The amount of the residual test substance was measured with following apparatus and conditions.

1)Apparatus

High performance liquid chromatograph mass spectrometer(LC/MS/MS) : UPLC Q-Premier XE(No.1)

Workstation : MassLynx 4.1

High performance liquid chromatograph (HPLC) : ACQUITY UPLC

Pump : Binary Solvent Manager

Auto injector : Sample Manager

Mass spectrometric detector (MSD) : Quattro Premier XE

2)Conditions

Column : VanGuard™ Pre-column ACQUITY UPLC BEH C18,
1.7 μ m, 2.1 mm i.d.× 5 mm, Waters Corp.
+ ACQUITY UPLC BEH C18, 1.7 μ m, 1.0 mm i.d.× 150 mm

Mobile phase :A1 : 20 mM ammonium formate aqueous solution : formic acid =
1000 : 1
B1 : methanol

Gradient conditions

Time	A1	B1
0.00 min	45%	55%
2.50 min	45%	55%
3.00 min	10%	90%
3.50 min	45%	55% (step gradient)
4.50 min	STOP	

Measurement time : 4.0 min

Flow rate : 0.12 mL/min

Injection volume : 1 μ L

Oven temperature : 60°C

Injection mode : Partial Loop With Needle Overfill

[MSD conditions]

Ion mode	: Electrospray Negative
Detection mode	: MRM
Monitoring ion	: Parent ion m/z 328.87 ([M-H] ⁻)
	Daughter ion m/z 284.80
Voltage	: Capillary 1.00 kV
	Cone 10 V
	Collision energy 14.0 eV
Temperatures	: Source Temp 100 °C
	Desolvation Temp 300 °C
Gas Flow	: Desolvation 500 L/hr
	Cone 50 L/hr
	Collision Gas Flow 0.10 mL/min

3) Calibration curve

The test substance was dissolved in purified water at a concentration of 100 mg/L to make a stock solution, which was further diluted with purified water to prepare standard solutions of 0.0500, 0.100 and 0.200 mg/L. Purified water was used as 0 mg/L standard solution. The standard solutions were analyzed with LC/MS/MS. The peak area of the test substance was plotted against the concentration.

The correlation coefficient was calculated to be 1.00 by the least squares method showing good linearity and the calibration curve was regarded passing through the origin. Amount of the test substance for each bottle was calculated from ratio of peak area for the sample to that for the standard solution of 0.200 mg/L. [Figure 2 and 3]

4) Detection limit

The minimum detectable peak area in the LC/MS/MS chromatogram was set at the peak area equivalent to 5% of standard solution of 0.200 mg/L. The detection limit of the test substance was calculated to be 1.5 mg in the recovery test and 1.6 mg in the measurement of residual test substance, respectively. Both values were based on the amount of the test substance in test bottles corresponding to this area. [Figure 3 and 4]

5) Recovery test

Duplicate test bottles identical to the test bottles 3-5 and a control bottle identical to the bottle 2 described in §2.1.1 were kept in closed system oxygen consumption measuring apparatus for 30 minutes.

Each test solution was treated by the procedures described in §2.1.3. The amount of the test substance was measured with LC/MS/MS. The recoveries of the test substance were 96 and 98%. The amount of the residual test substance measured for bottles 3-6 were corrected with the average recovery of 97%. In bottle 2, the amount of the residual test substance was confirmed to be below the detection limit. [Table 3 and Figure 4]

2.2 Evaluation of test results

2.2.1 Equations for calculation of degradability

Degradabilities based on measured values of the BOD, DOC and residual substance amount were calculated by the following formulas:

1) Degradability based on the BOD

$$\text{Degradability (\%)} = (\text{BODs} - \text{BODb}) / \text{ThOD} \times 100$$

where BODs : Oxygen consumption (mg) in bottles 1, 3, 4 or 5

BODb : Oxygen consumption (mg) in bottle 2

ThOD : Theoretical oxygen demand (mg) of aniline or the test substance

ThOD

aniline : $\text{C}_6\text{H}_7\text{N} + 35/4\text{O}_2 \rightarrow 6\text{CO}_2 + 7/2\text{H}_2\text{O} + \text{NO}_2$

ThOD : 90.2 mg O₂/30.0 mg-aniline

test substance : The test substance were assumed to be mineralized as shown below:

$\text{C}_6\text{HO}_3\text{F}_{11} + 19/2\text{O}_2 \rightarrow 6\text{CO}_2 + 1/2\text{H}_2\text{O} + 11\text{F}$

ThOD : 27.6 mg O₂/30.0 mg-test substance

2) Degradability based on the DOC

$$\text{Degradability (\%)} = [1 - (\text{DOCs} - \text{DOCb}) / \text{DOCc}] \times 100$$

where DOCs : DOC (mg) in bottles 3, 4 and 5

DOCb : DOC (mg) in bottle 2

DOCc : DOC (mg) in bottle 6

3) Degradability based on the residual amount of test substance

$$\text{Degradability (\%)} = (1 - \text{Cs} / \text{Cc}) \times 100$$

where Cs : amount (mg) in bottles 3, 4 and 5

Cc : amount (mg) in bottle 6

2.2.2 Judgment of degradability

The degradability of the test substance is comprehensively judged from measurement values and degradability results based on the BOD, the DOC and the residual test substance amount.

2.2.3 Validity

The test is valid in the case that the results fulfill all of following criteria.

- 1) The degree of degradability based on the BOD measurement of reference substance (aniline) is greater than 60% after 14 days.
- 2) The difference of extremes of replicate values of degradability at day 28 is less than 20%.
- 3) The BOD in the blank (bottle 2) is less than 18 mg O₂ after 28 days.

3 RESULTS

3.1 Factors which might have affected the reliability of the test results

There was no specific factor which might have affected the reliability of the test results.

3.2 Observation of the contents after exposure period

The color of the solution and growth of the sludge in the test bottles were observed in contrast with the control (bottle 2).

The solution in bottle 1 was white, solution in bottles 2, 3, 4, 5 and 6 was colorless.

Growth of the sludge was observed in bottle 1 whereas no growth of the sludge was observed in bottles 3, 4 and 5.

3.3 pH measurement

After 28 days of exposure, pH values were all 7.3 for bottles 3, 4 and 5, and 3.6 for bottle 6, respectively. [Table 1]

3.4 Degradability based on the BOD

The BOD^{*1} in bottles 3, 4, and 5 (as corrected with the value in bottle 2) were 1.1, -0.3 and 1.6 mg, respectively, and the BOD in bottle 6 was 0.0 mg.

(*¹ Maximum theoretical value = 27.6 mg)

The degradabilities based on the BOD measurement were calculated to be 4, 0 (Calculated value was -1) and 6 % for bottles 3, 4, and 5, respectively.

[Table 1 and Figure 5]

3.5 Degradability based on the DOC

The DOC^{*2} in bottles 3, 4 and 5 (as corrected with the value in bottle 2) were 6.3, 6.2 and 6.6 mg, respectively, and the DOC in bottle 6 was 6.4 mg.

(*² Initial amount = 6.5 mg)

The degradabilities based on the DOC were calculated to be 2, 3, and 0 (Calculated value was -3) % for bottles 3, 4 and 5, respectively.

[Table 1 and 2]

3.6 Degradability based on the residual test substance amount

The amounts of residual test substance^{*3} were 29.3, 30.9 and 29.8 mg in bottles 3, 4 and 5, respectively and 30.7 mg in bottle 6.

(*³ Initial amount = 30.0 mg)

The degradabilities based on the residual test substance amount were calculated to be 5, 0 (Calculated value was -1), and 3% for bottles 3, 4, and 5, respectively.

[Table 1, 4 and Figure 6]

3.7 Validity of the result

The biodegradability test was judged valid since the measurement results fulfilled all of the validity criteria of the guideline.

4 DISCUSSION

From the degradability results based on the BOD (av. 3%), the degradability results based on the DOC (av. 2%) and the residual test substance amount (av. 3%), it is concluded that the test substance is not readily biodegradable under the conditions of this test.

Table 1 Summary of the test results**a) BOD measurement, pH**

Bottle No.	Sample description	BOD(mg)				pH	
		day 7	day 14	day 21	day 28	day 0	day 28
6	Water + test substance	0.0	0.0	0.0	0.0	3.5	3.6
3	Activated sludge + test substance No.1	2.1	3.6	5.7	6.6	7.0	7.3
4	Activated sludge + test substance No.2	2.0	3.2	4.7	5.2	7.0	7.3
5	Activated sludge + test substance No.3	2.4	4.1	6.1	7.1	7.0	7.3
1	Activated sludge + Aniline	50.1	67.0	72.1	73.8	---	8.0
2	Control blank	1.3	2.5	4.7	5.5	7.0	7.2

b) Measured values (day 28)

		Activated sludge + test substance			Water + test substance	Theoretical value
		No.1	No.2	No.3		
		Bottle No.3	Bottle No.4	Bottle No.5	Bottle No.6	
BOD ^{*1}	mg	1.1	-0.3	1.6	0.0	27.6
DOC ^{*1}	mg	6.3	6.2	6.6	6.4	6.5
Test substance	mg	29.3	30.9	29.8	30.7	30.0
(LC/MS/MS)	%	98	103	99	102	—

*1 : Value of [Activated sludge + test substance] is corrected with BOD or DOC value of [Control blank].

c) Degradabilities (%)

		Activated sludge + test substance			Average
		No.1	No.2	No.3	
		Bottle No.3	Bottle No.4	Bottle No.5	
BOD	%	4	0(-1) ^{*2}	6	3
DOC	%	2	3	0(-3) ^{*2}	2
Test substance	%	5	0(-1) ^{*2}	3	3

*2 : Where % degradability is calculated to be negative, this value is shown in parentheses.

Table 2 Result of DOC measurement

	Bottle 2		Bottle 3		Bottle 4		Bottle 5		Bottle 6	
	TC		TC		TC		TC		TC	
	area	mgC/L	area	mgC/L	area	mgC/L	area	mgC/L	area	mgC/L
Measure 1	755	1.855	9389	23.070	9253	22.740	9786	24.050	8616	21.170
Measure 2	741	1.821	9394	23.080	9252	22.730	9804	24.090	8752	21.510
Measure 3	744	1.828	9385	23.060	9284	22.810	9823	24.140	8812	21.650
Mean	747	1.835	9389	23.070	9263	22.760	9804	24.093	8727	21.443

Calibration curve	
TC standard (area)	
40 mgC/L	15918
80 mgC/L	32256
	16040
	32334
	16162
	32365
	16040
	32318

	IC		IC		IC		IC		IC	
	mgC/L		mgC/L		mgC/L		mgC/L		mgC/L	
	area	mgC/L	area	mgC/L	area	mgC/L	area	mgC/L	area	mgC/L
Measure 1	0	0.000	0	0.000	0	0.000	0	0.000	0	0.000
Measure 2	0	0.000	0	0.000	0	0.000	0	0.000	0	0.000
Measure 3	0	0.000	0	0.000	0	0.000	0	0.000	0	0.000
Mean	0	0.000	0	0.000	0	0.000	0	0.000	0	0.000

IC standard (area)	
0 mgC/L	0
10 mgC/L	4266
	0
	4356
	0
	4283
	0
	4302

TOC-5000A Conditions

	TC (mgC/L)	IC (mgC/L)	TOC (mgC/L)	TOC (mg)
Bottle 2	1.835	0.000	1.835	0.6
Bottle 3	23.070	0.000	23.070	6.9
Bottle 4	22.760	0.000	22.760	6.8
Bottle 5	24.093	0.000	24.093	7.2
Bottle 6	21.443	0.000	21.443	6.4

TC Furnace Temp. : 680°C
 TC Catalyst : Normal
 Syringe Size : 250 μ L
 Measurement Mode : TOC
 Air flow rate : 150 mL/min
 No of Injects : 3
 Range : $\times 5$
 Inj vol : 30 μ L

Table 3 Calculation of recovery and detection limit

	A	B	C	D	E
	Peak area count	Recovered amount mg	Added amount mg	Recovery %	Detection limit mg
Recovery test 1	2351.2	28.7	30.0	96	---
Recovery test 2	2399.8	29.3	30.0	98	---
Blank	<122.7	<1.5	0.0	---	1.5

Average recovery: 97 %

Standard solution

Concentration

F: 0.200 mg/L

Volume of test solution in bottle

Peak area

G: 2454.4 count

Dilution rate

H: 0.3 L

I: 500

Equation : $B = F \times A \div G \times H \times I$ D = $B \div C \times 100$ E was derived by rounding B up to first decimal place.

Table 4 Residual test substance amount in test bottles

Bottle No.	A	B
	Peak area count	Test substance in bottle mg
2	<79.7	<1.6
3	1511.3	29.3
4	1590.9	30.9
5	1536.2	29.8
6	1583.9	30.7

Standard solution

Concentration

C: 0.200 mg/L

Volume of test solution in bottle

Peak area

D: 1594.0 count

Average recovery

Dilution rate

E: 0.3 L

F: 500

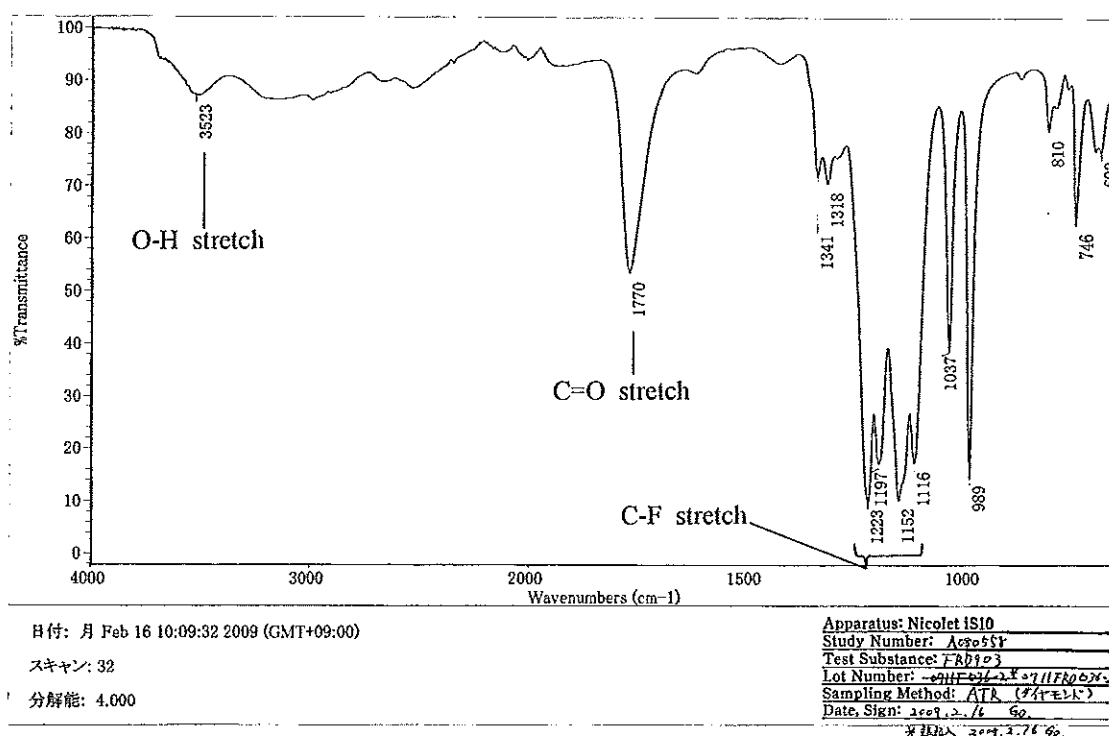
G: 97 %

Equation : $B = C \times A \div D \times E \times F \div G \times 100$

Detection limit: 1.6 mg

Figure 1 Infrared absorption spectra of the test substance

Before the exposure period



After the exposure period

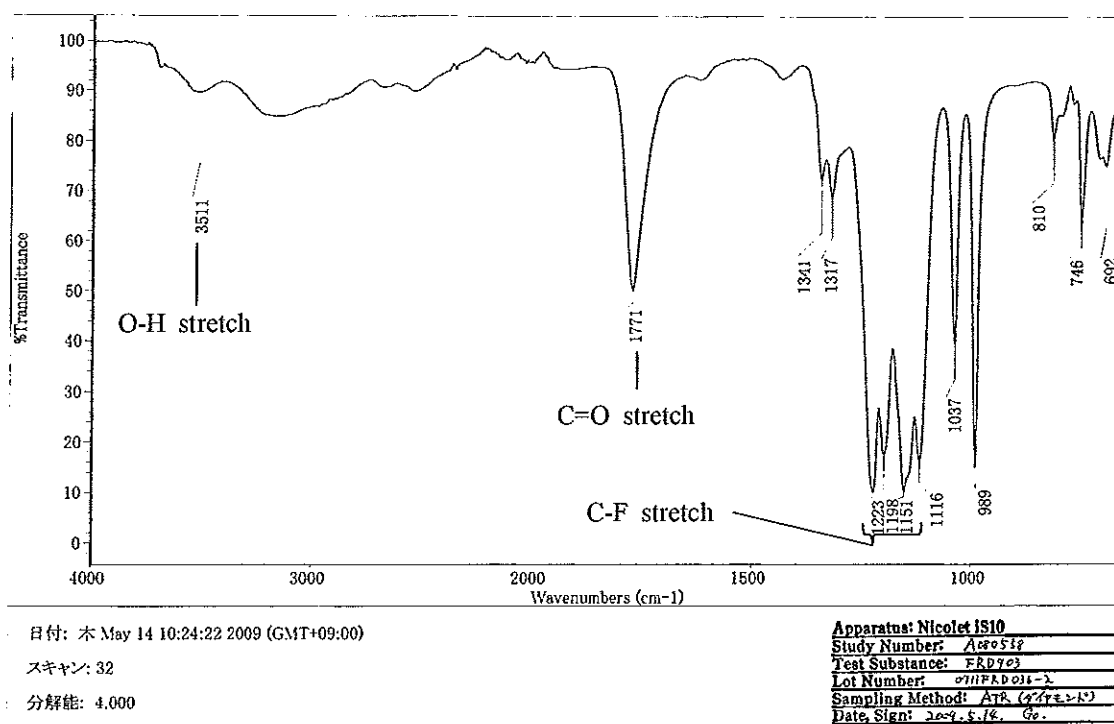


Figure 2 Calibration curve of the test substance

Curve Fitting [Least Square Method]

Input Data		
No.	Concentration X (mg/L)	Peak Area Y (count)
1	0	0
2	0.0500	723.7
3	0.100	1340.3
4	0.200	2757.8

$$Y = 5.9800E+00 + 1.3708E+04 \times X$$

$$r = 0.99969$$

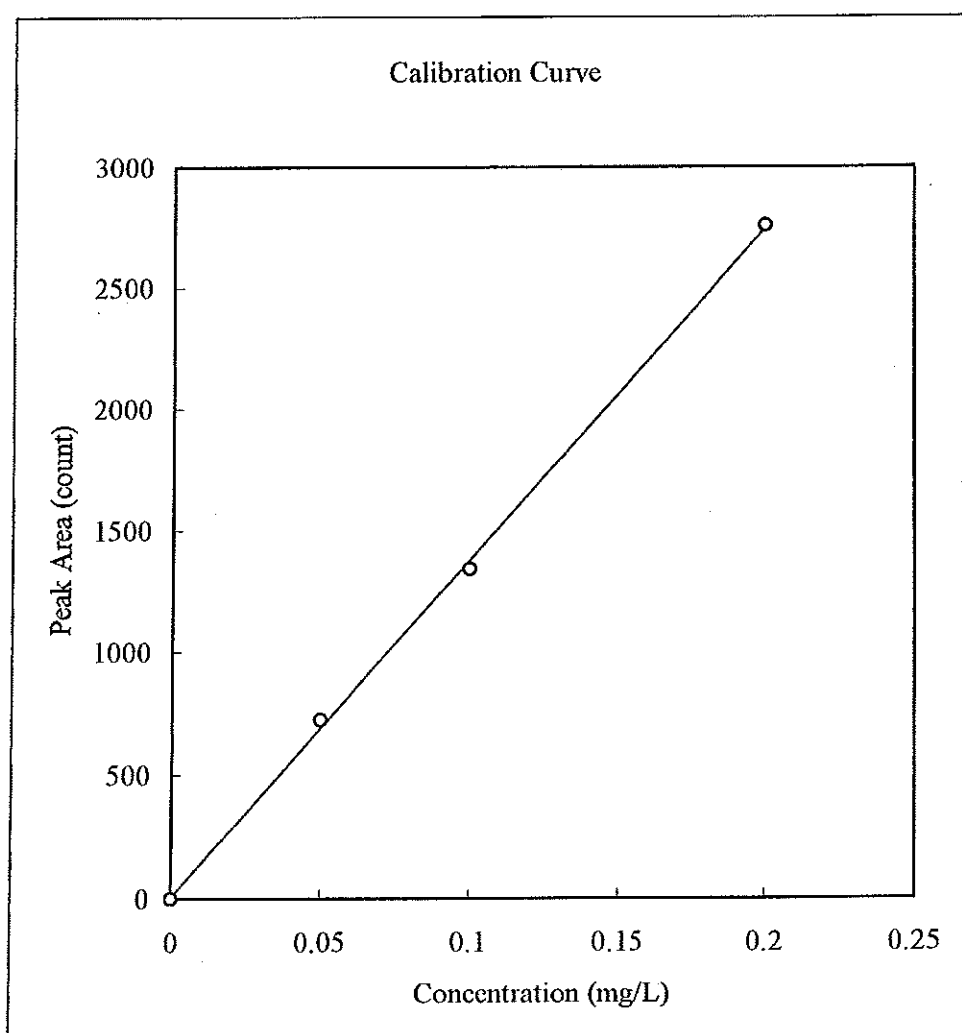
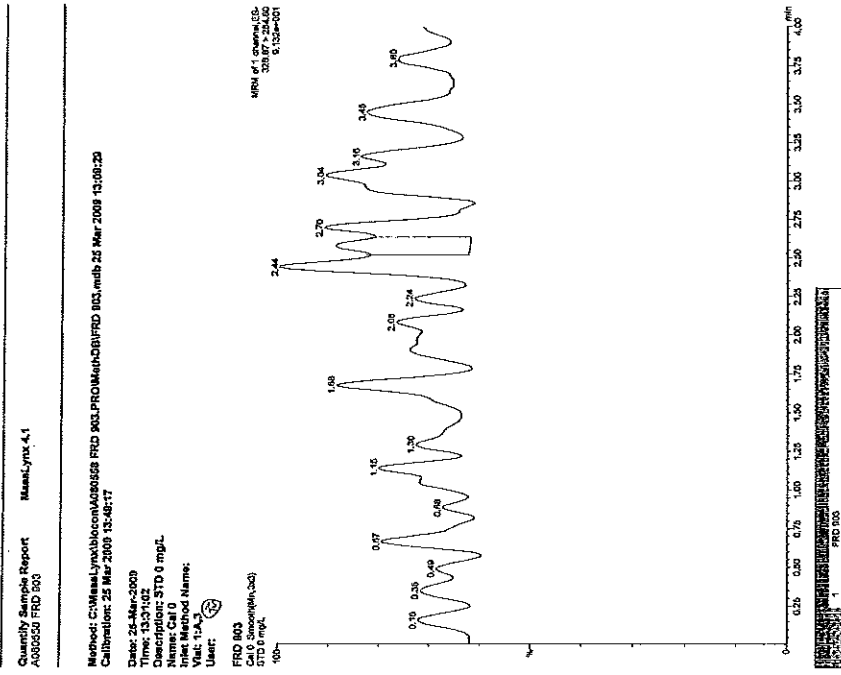


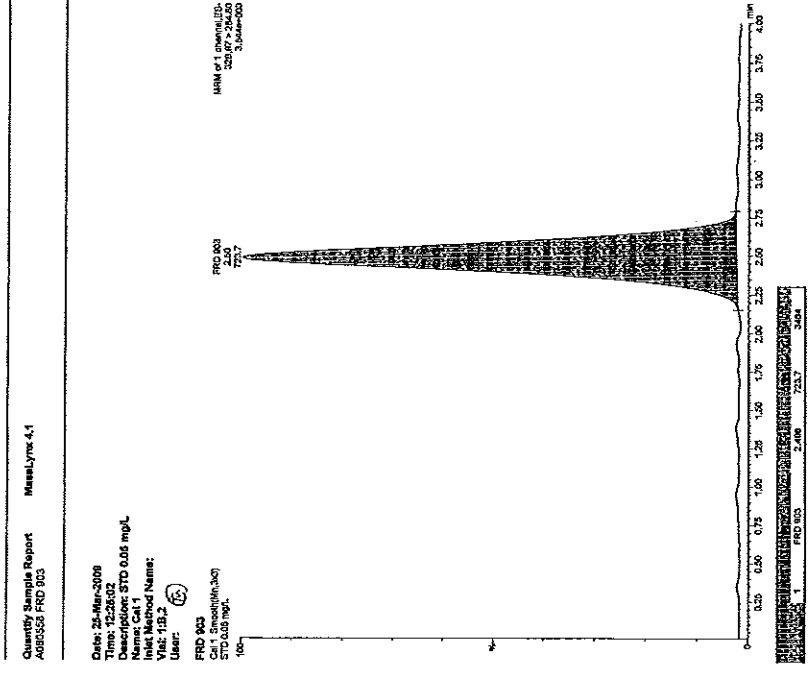
Figure 3 LC/MS/MS chromatograms of the test substance---Calibration curve

0 mg/L standard solution



試験名: FRD 903 の分解試験
試験番号: A080558
測定日: 2009.3.25
測定者: (S)

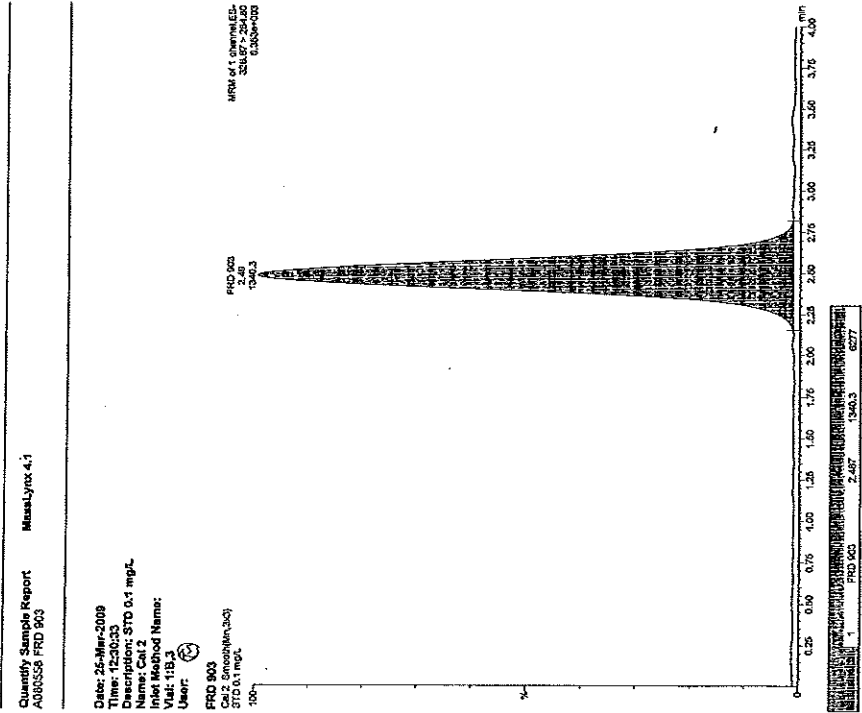
0.0500 mg/L standard solution



試験名: FRD 903 の分解試験
試験番号: A080558
測定日: 2009.3.25
測定者: (S)

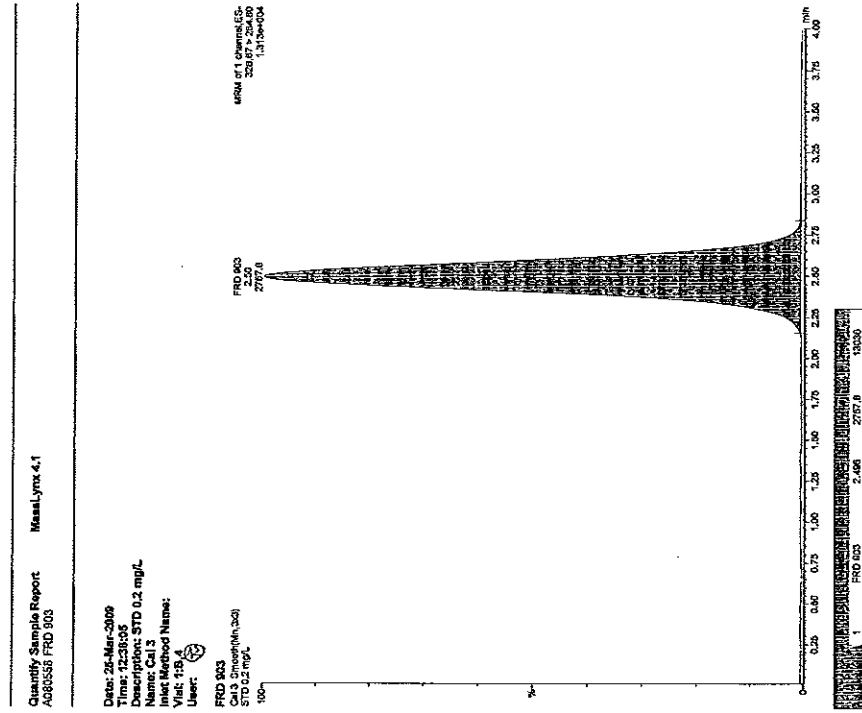
Figure 3 Continued

0.100 mg/L standard solution



試薬名: FRD 903	の分標値試験
試薬番号: A080558	
測定日: 2009.3.25	測定者:

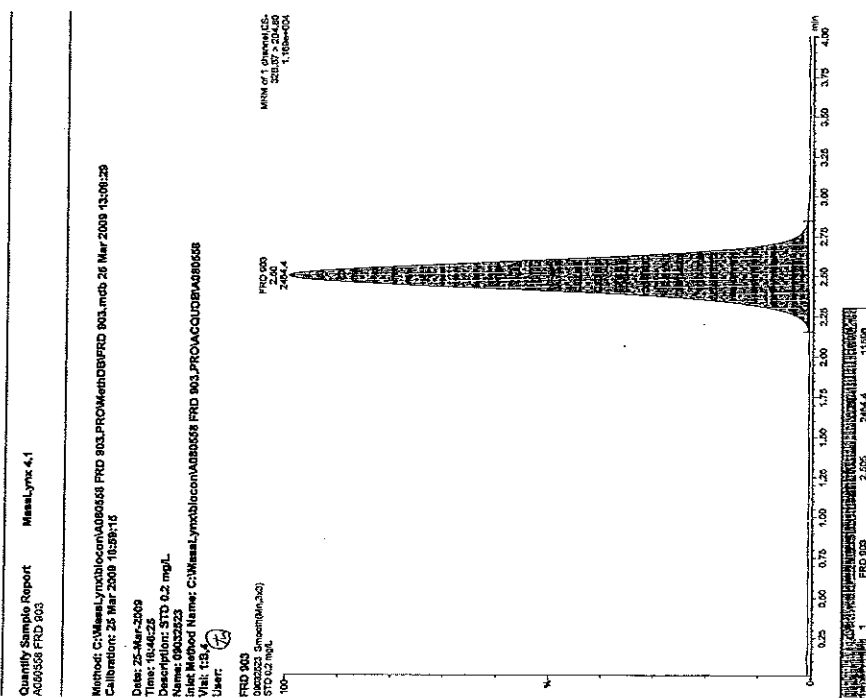
0.200 mg/L standard solution



試薬名: FRD 903	の分標値試験
試薬番号: A080558	
測定日: 2009.3.25	測定者:

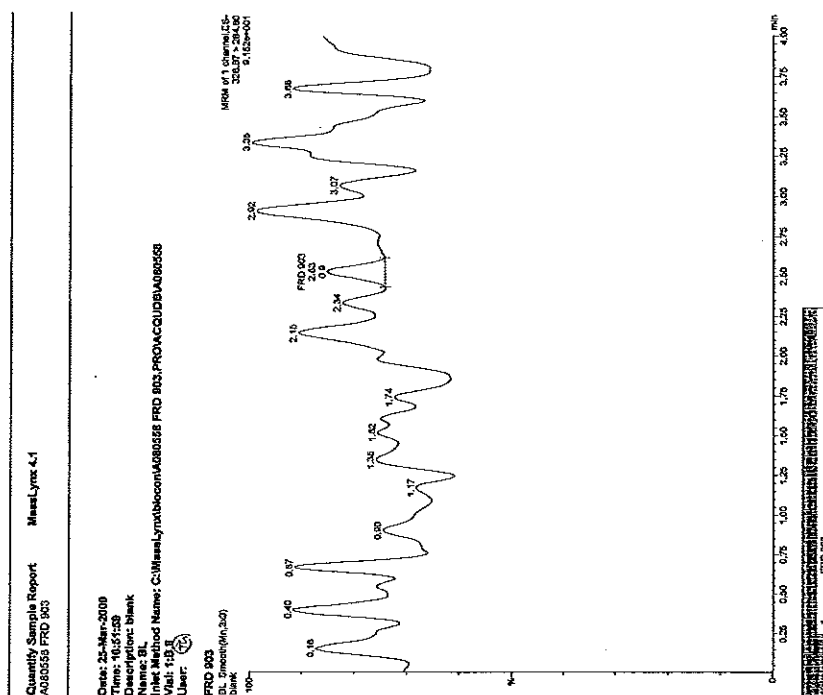
Figure 4 LC/MS/MS chromatograms of the test substance---Recovery and detection limit

0.200 mg/L standard solution



検査名: PRD 903 0分程度試験
試験番号: A080558
測定日: 2009.3.25 測定者: [Signature]

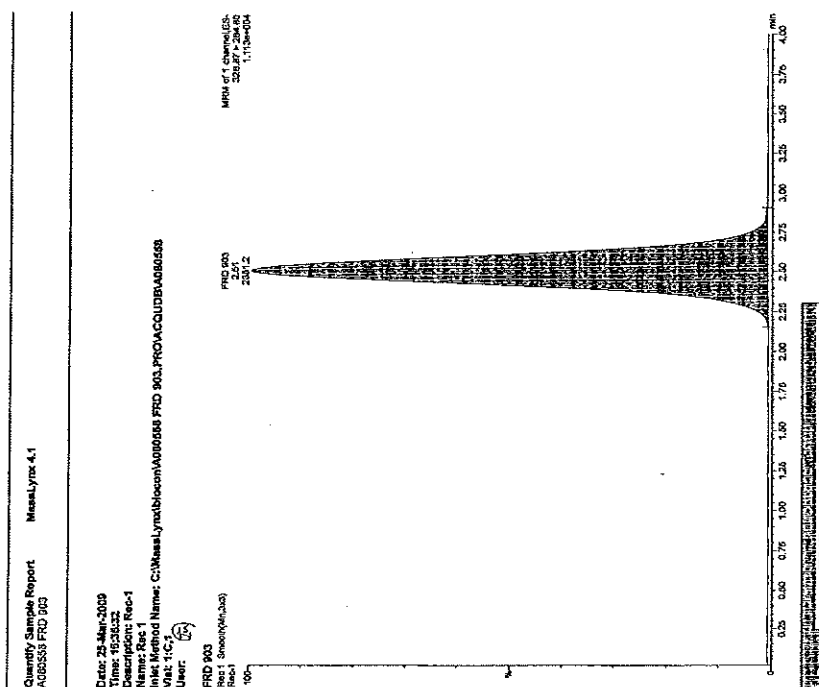
Blank test



検査名: PRD 903 0分程度試験
試験番号: A080558
測定日: 2009.3.25 測定者: [Signature]

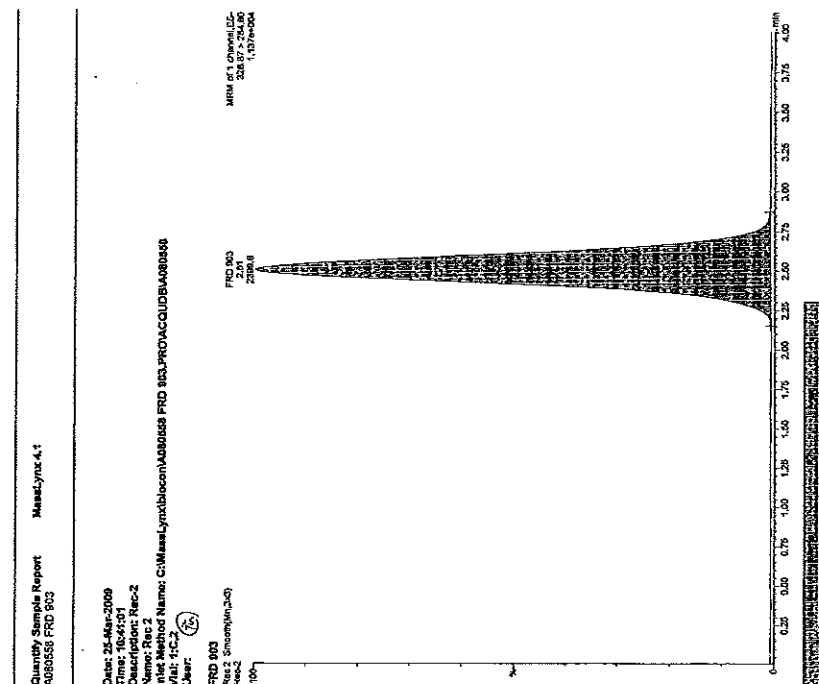
Figure 4 Continued

Recovery test 1



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測定日: 2009.3.25 測定者: (S)

Recovery test 2



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試験番号: A080558
測定日: 2009.3.25 測定者: (S)

Figure 5 BOD chart

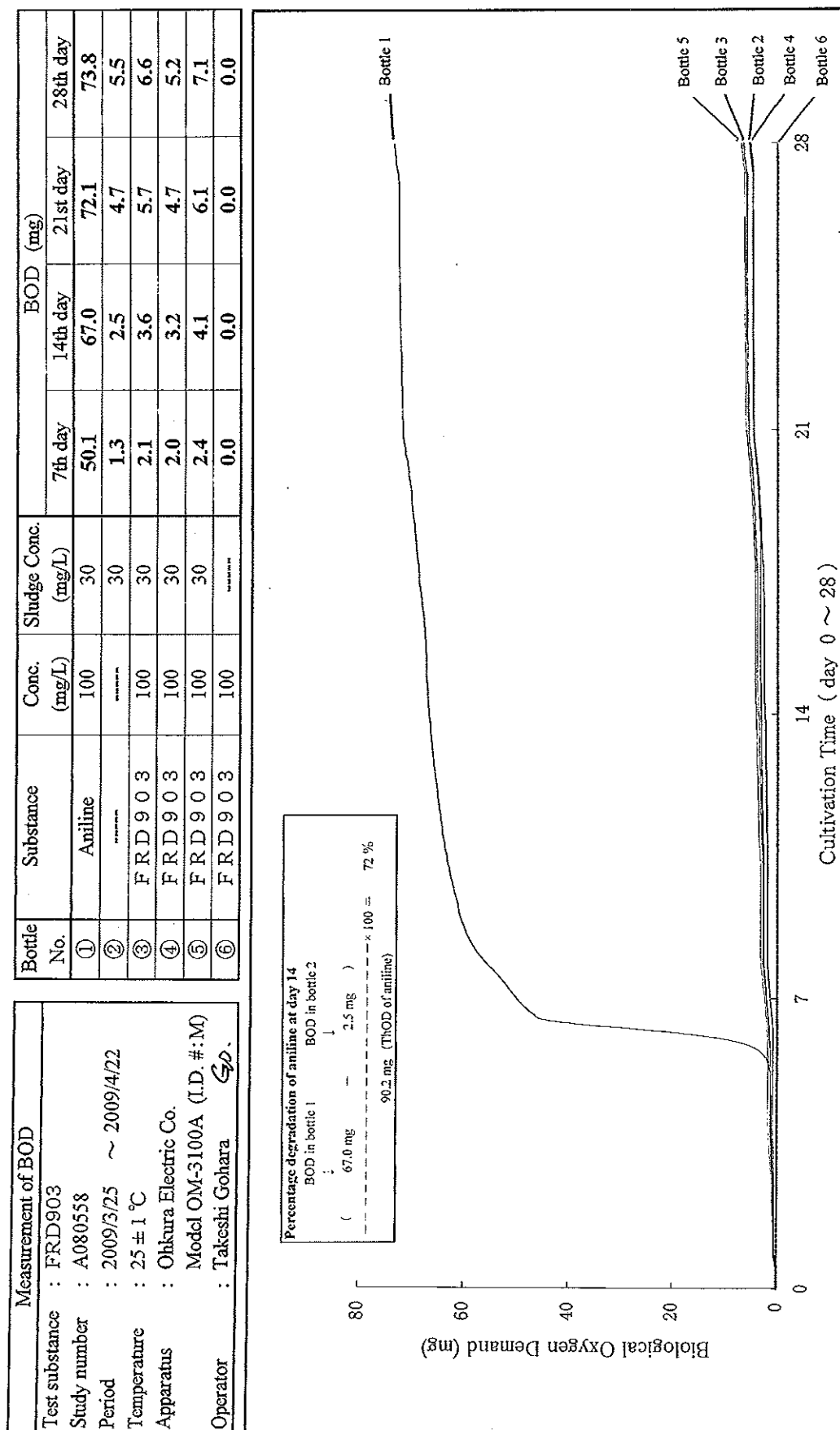
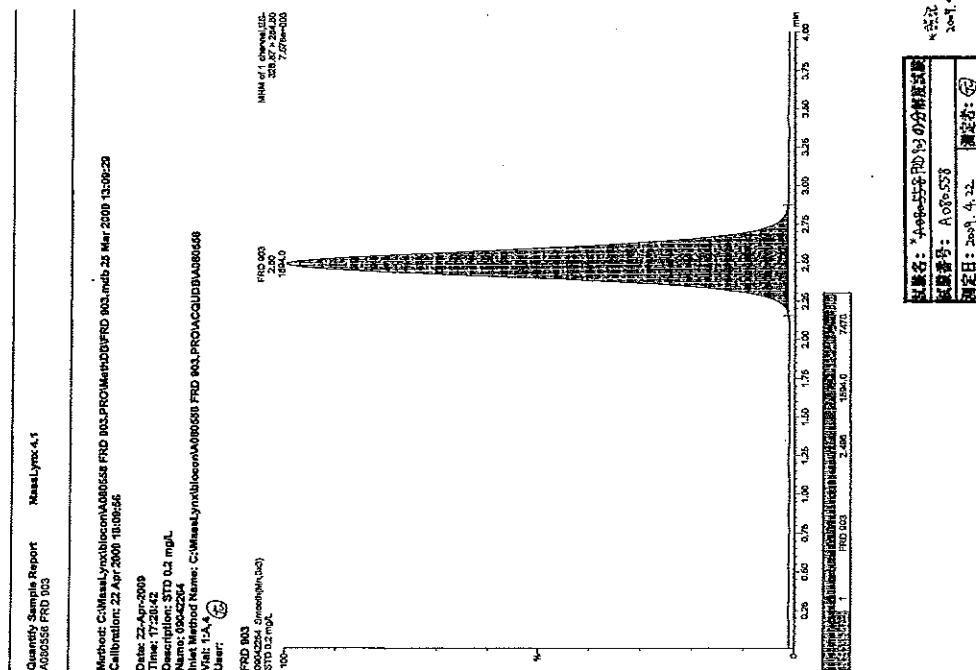


Figure 6 LC/MS/MS chromatograms of the test substance—Measurement of residual test substance amount

0.200 mg/L standard solution



Bottle 2 (blank test of sludge)

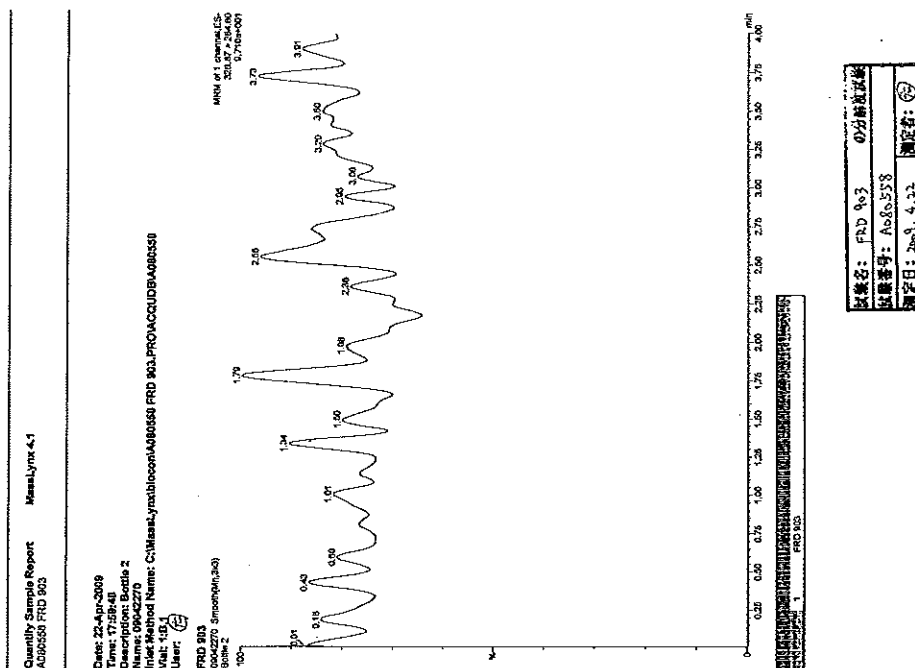
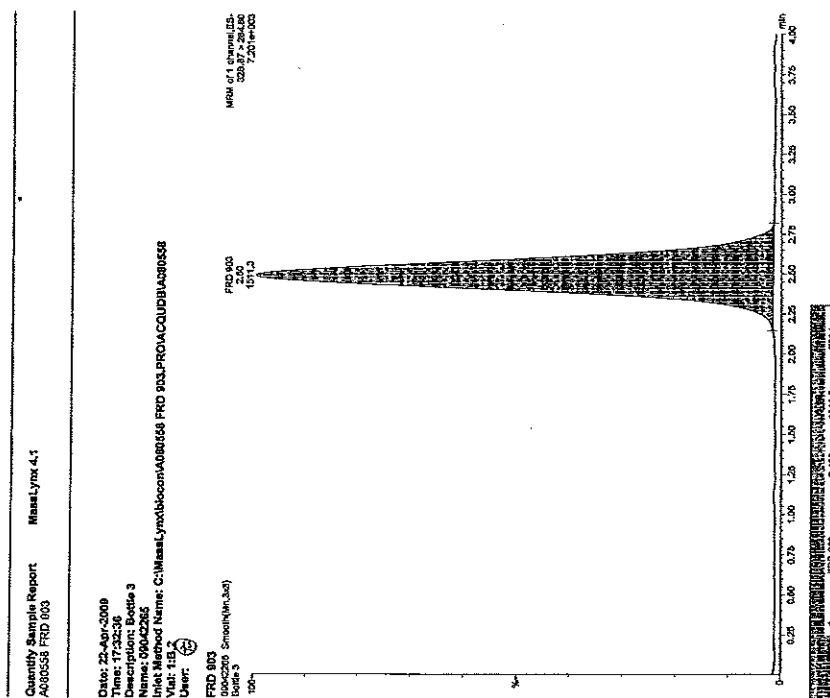


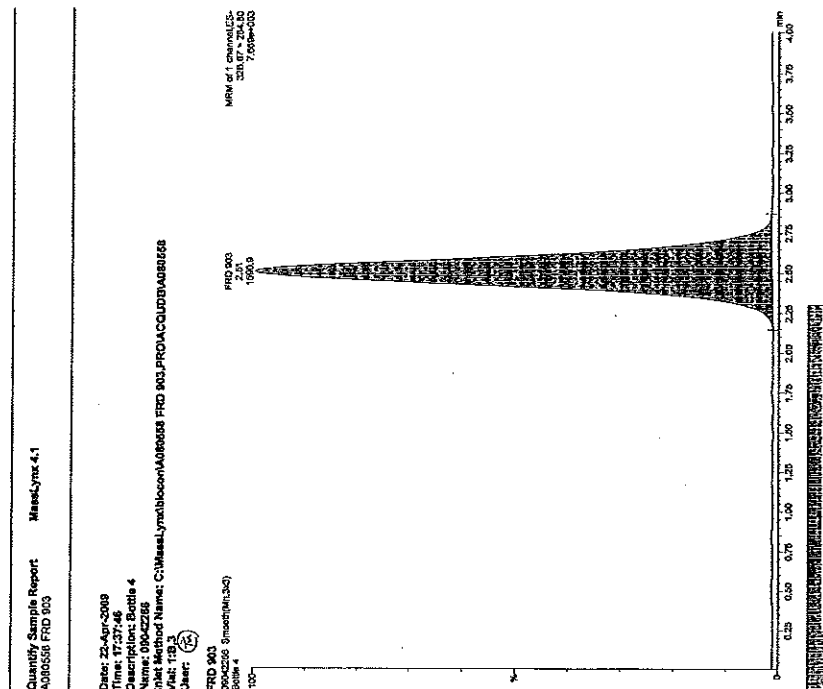
Figure 6 Continued

Bottle 3 (sludge + test substance)



試料名: PRD 903 の分析試料
試料番号: A080558
測定日: 2009.4.22 測定者: (S)

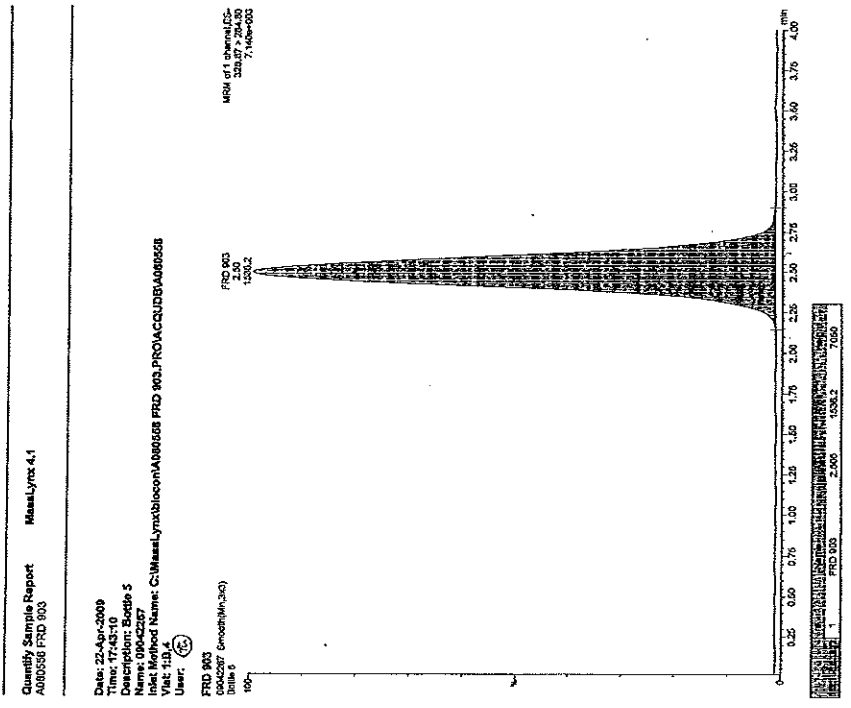
Bottle 4 (sludge + test substance)



試料名: PRD 903 の分析試料
試料番号: A080558
測定日: 2009.4.22 測定者: (S)

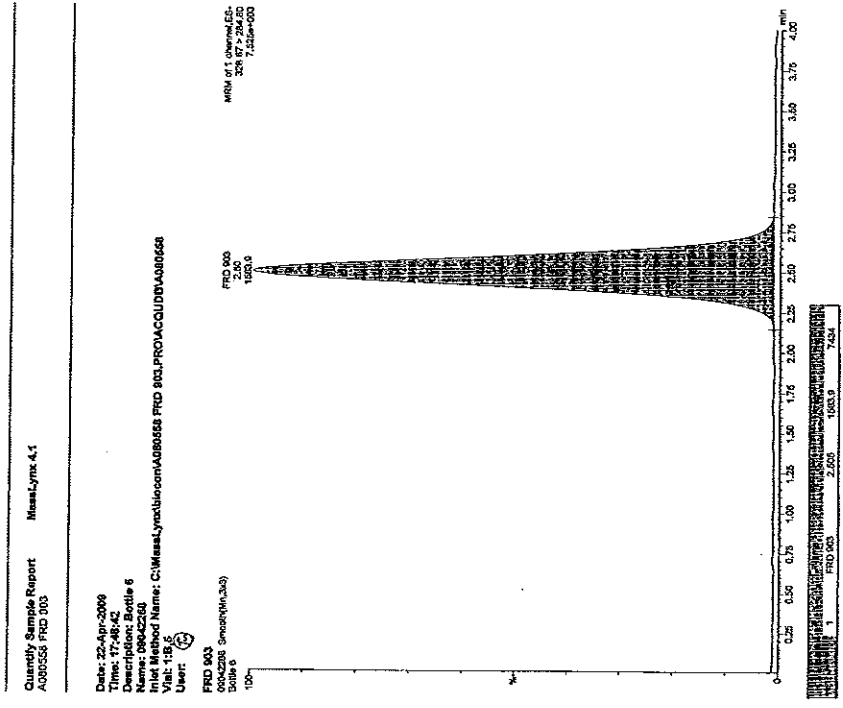
Figure 6 Continued

Bottle 5 (sludge + test substance)



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測定日:	2009. 4. 22	測定者: (C)

Bottle 6 (water + test substance)



試料名:	PRD 903	の分析結果表
試料番号:	A080558	
測定日:	2009. 4. 22	測定者: (C)